Communication.

AN EFFICIENT STIRRING APPARATUS.

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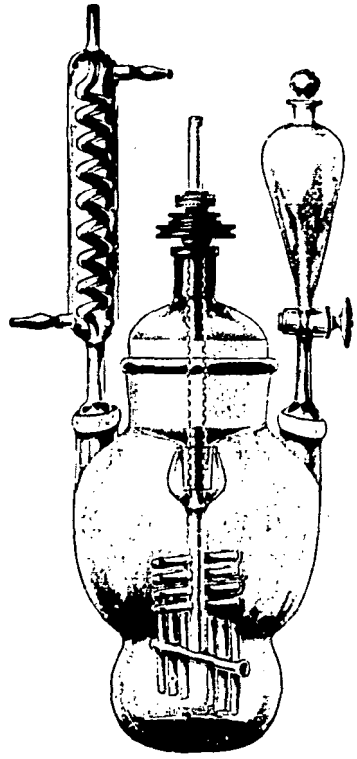
(Contribution from the Davy-Faraday Laboratory, Royal Institution.)

In chemical literature many suggestions are given for stirring apparatus which can be made by the worker himself from the simplest materials. Modern laboratory work, however, does not afford time for making apparatus: everything must be ready to hand and certain to work. Moreover, no ready-made apparatus would stir under varying conditions, such as under reflux, with exclusion of air, at high temperatures, etc. We had thus to make such an apparatus for ourselves.

The main idea of the construction is to meet as many needs as possible with *one* apparatus. We therefore made a constriction in the vessel, so that the part below the constriction held 500 c.c., in order to secure effective stirring of small quantities of liquid down to 150 c.c. The total capacity of the flask is about 1500 c.c., and this volume of liquid can be easily worked.

We had to avoid all rubber and cork stoppers, so as to be able to use the apparatus for nitration and similar processes, a thing which, so far as we know, is not done by any previous contrivance.* It was also necessary to make it possible to heat the whole arrangement in an oil- or water-bath, which, in spite of the strong glass used in its construction, can be done without any risk, owing to the high efficiency of present-day glass manufacture.



The apparatus also must easily be taken to pieces. This is done by loosening the screw above the driving-wheel. The inside screw is split, and, on screwing down the nut on to it, contact is made between the metal and the stirring-rod. The pear-shaped vessel fused to the shaft of the stirring rod serves the purpose—by trapping the lower end of the ground stopper in a fluid seal—of effectively preventing contact between the vapours in the main flask and the metal parts of the apparatus. In order to make all parts of the apparatus detachable, we had to arrange this pear-shaped vessel inside the main flask in which the reaction takes place.

The shape of the stirrer (which reaches nearer to the bottom than is shown in the diagram) adapts it for stirring up precipitates and making oils into emulsions.

The size of the hollow ground-stopper, which was necessitated by that of the stirrer, at first seemed somewhat dangerous, but nevertheless proved quite satisfactory. We chose this form of stirrer (which corresponds to the form used by Pieraerts (Chem. Zeit. 29, 671 (1905)), and which is like that used in the mash-tubs of breweries) because those with movable arms,* used, for instance, by E. Fischer (*loc. cit.*), often give trouble, especially when dealing with heavy liquids or thick precipitates.

Working at ordinary temperatures, it is sufficient to fill the pear-shaped vessel round the stirrer with water, paraffin oil, or mercury (in the latter case only half full). There is no danger of these liquids coming over the edge. At higher temperatures, some of the reaction mixture will distil over into the pear-shaped jacket. If interaction with the mercury should take place, as, for instance, in nitration, it is better to use instead a liquid heavier than water or dilute nitric acid, such as nitrobenzene or bromnaphthalene, etc., or, better still, to fill the jacket nearly full of water, then set up the whole apparatus, raise the stirrer a little, and, with a pipette, introduce a little paraffin oil into the space thus left between the metallic connection attached to the stirrer and the connection in the neck of the hollow stopper. The oil collects in the elongated joint of the ground-stopper, above the water, which may be contaminated with acid constituents, and prevents these from coming into contact with the metal. Our experience shows that even in rapid rotation the oil always stays in its place inside the elongated part of the stopper, but it is necessary to find out what quantity of paraffin is needed, as the different forces produce a stretching of the oil-layer, which may cause oil to enter the jacket itself.

In cases where contamination by acid vapours may be expected, the apparatus is very useful. When working in an atmosphere other than air, *e.g.* hydrogen, the dropping-funnel can be replaced by a stopper fitted with inlet- and outlet-tubes, and liquids can be introduced through the condenser. For purposes of temperature measurement, the apparatus is also fitted with an upright condenser into which a thermometer can be inserted.

The apparatus stands firm by reason of its own weight, *one* clamp being sufficient, and even in rapid rotation the vibration is very slight. Steady rotation is best maintained by means of a small electro-motor, and the speed is regulated by tightening or loosening the belt connecting the motor to the stirrer.

Care should be taken to oil the joint of the stirrer and to grease the ground-stopper well. If grease must be avoided, graphite can be used instead. (R. Kempf, J. Pr. Chem. (2), **78**, 207 (1908).)

After continued working at high temperature, it is advisable to loosen the stopper while the apparatus is still warm. We never experienced any difficulty in doing this, as the grinding is very carefully carried out. We kept the apparatus working for days at 100°, being placed on a small porcelain support covered with sacking in a vessel of boiling water.

Among the numerous experiments for which this apparatus was successfully used, only the almost quantitative oxidation of *o*-nitrotoluene to *o*-nitrobenzoic acid may be cited, which Ullmann (Ber. 36, 1797 (1903)) carried out in a specially constructed copper kettle.

The apparatus may be obtained from Gebr. Muencke, Schumannstr. 2, Berlin, N.W.

* E. Fischer. Anl. zur Darstellung organ. Präparate, 7 Aufl, 1905, 23, Ber., **41**, 2882 (1908); Brühl. Ber., **37**, 923 (1904). Hesse. Ber., **39**, 1149 (1906), etc.

* Schulze, Ber., **29**, 2883 (1896); Mittelbach, Chem. Zeit., **31**, 584 1907). Plaeln, Chem. Zeit., **37**, 691 (1913).